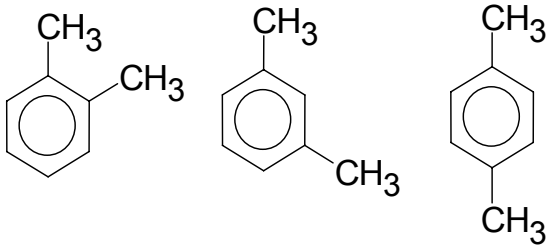
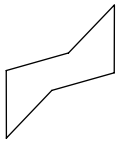
Experiment 6. QUANTINTATIVE ANALYSIS OF XYLENES USING IR SPECTROSCOPY

Materials

1. Numbered vial containing your assigned unknown mixture of xylenes.
2. A bottle of spectral grade cyclohexane.
3. A 2.0 ml syringe for loading sealed infrared cells.
4. A 0.1 mm sealed IR cell. These cells are kept in a dessicator!
5. Samples of pure o-, m-, and p-xylenes.



o-xylene m-xylene p-xylene



cyclohexane

I. Introduction

The purpose of this experiment is to analyze a mixture of xylene isomers quantitatively for its ortho, meta and para isomers. The IR spectra of the pure compounds are found in Atlas so you know which peaks are associated with each isomer. The concentrations of **o-, m- and p-xylenes** will be determined by application of a modified version of the baseline technique to the unknown and standards.

II Sealed Cells.

The sealed cells used in this experiment have a very small path length (often 0.10 mm), are quite fragile and must be handled carefully, particularly during the filling and emptying operations. The cells are filled by using a syringe to introduce the sample.

The sealed cells should always be rinsed and dried when your work with them is done. NEVER rinse with water! Remember these cells are sodium chloride. Rinse with a dry organic solvent which evaporates quickly such as $CHCI_3$.

III. Procedure

A. Preparation of Solutions.

1. Pipette the following volumes of o-, m- ,and p-xylenes into a DRY 50 mL volumetric flask. The flask cannot be wet or you will get a 2-phase system instead of a solution!

1. 1.00 ml m-xylene
2. 1.00 mL p-xylene
3. 1.00 mL o-xylene
4. 0.40 ml m-xylene + 0.40 mL p-xylene + 0.40 mL o-xylene
5. 0.20 ml m-xylene + 0.20 mL p-xylene + 0.20 mL o-xylene

Dilute to the mark with spectral grade cyclohexane.

B. Running the Spectra.

First, determine the thickness of the cuvet you use. Count the number of fringes N and the location of the starting ν_1 and ending maximum ν_2 (you start from No.0, not from No.1 !)

$$l(cm) = \frac{N}{2 \times (\nu_1 - \nu_2)}$$

After that, proceed to **absorption measurements**.

l .Fill a 0.10 mm sealed sample cell with pure solvent (cyclohexane) using a syringe and insert the Teflon plugs to prevent loss of solution. Obtain the background spectrum using the FTIR spectrophotometer. Re-range the instrument and scan the range 12 to 16 micrometers (860 to 600 cm^{-1}) with 1 cm^{-1} resolution. Empty the sample cell and rinse with a syringe full of the solvent. Dry the cell.

2. Refill the clean, dry sample cell with solution #1 and run its spectrum.

3. Empty, rinse, dry and refill the sample cell with solution #2 and so on.

4. Dilute your sample with cyclohexane (1 mL of the sample into 25 mL flask). Run the spectrum of unknown sample. Process your data.

The values of $A=\log T_o/T$ for standard solutions are then plotted against concentration to give a Beer's Law plot. See your textbook for possible additional information on the baseline technique. Plot three calibration curves. Calculate molar absorptivities of for each xylene absorption band, and determine detection limits and determination limits for each compound.

Waste Disposal

When done with the experiment, empty your waste into the bottle in the hood labeled WASTE CYCLOHEXANE AND XYLENES.

IV. IR spectra modeling using HyperChem 7.0.

Using any computer in our computer laboratory, open HyperChem 7.0 program. Load appropriate file ("o-xylene", "p-xylene", etc). Model the IR spectra and compare with the experimental data. Take a look at animation of the vibrations.